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Fission Track Dating Using External Detector Method: a Laboratory Procedure

By

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Abstract

A fission track dating laboratory procedure using external detector method is presented for the most commonly used minerals, zircon, apatite and sphene. The procedure consists of separation of these minerals from the rock samples, their mounting, grinding/polishing, chemical etching, thermal neutron irradiation and counting of their fission tracks. It incorporates some technical refinements made in FTD laboratory of Kyoto University. A computer program to calculate zeta constants and FT ages is also given at the end together with the relevant formulas.

1. Introduction

Since its birth in the mid 1960s, fission track dating (FTD) has shown a rapid growth. It has been applied to various scientific fields ranging from astronomy to anthropology (FLEISCHER et al., 1975), and has been highlighted as a diagnostic tool for thermal history analysis (GLEADOW et al., 1986). For interlaboratory comparison of FTD data, two aspects namely the ambiguity of age calibration constants and the variety of dating procedure need attention. Zeta calibration approach proposed by HURFORD and GREEN (1982, 1983) is proving a useful attempt to resolve the problem of age calibration constants. As for the various dating procedures, GLEADOW (1981) has reviewed and compared them. Of the various alternatives, external detector method (EDM) is considered to be the most reliable and useful due to its following advantages:

1) Range of age determination is generally wide
2) Age is not affected by variation of uranium distribution within and between crystals
3) Contamination effect of detrital crystals can be assessed for dating of volcanic ash layers
4) Later check of sample is possible
5) Sample of small amount can be dated

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6) Precision of age determination is generally high.

The aim of this article is to present in detail the FTD laboratory procedure of EDM, incorporating some technical refinements, as practiced in FTD laboratory of Department of Geology and Mineralogy, Kyoto University.

2. Mineral Separation

For FTD of zircon, apatite and sphene, it is necessary, in general, to separate these minerals efficiently from their host rocks. They are separated from other minerals or rock fragments using their characteristic properties of size, magnetism and density. Before starting the separation procedure, it is sometimes convenient to examine the petrographic thin sections for assessing the presence of these minerals and their suitability for FTD. Mineral separation involves several steps as shown in the flow chart (Fig. 1).

2.1. CRUSHING

This procedure is indispensable to the mineral separation except soft or brittle samples, such as volcanic ashes. For sufficient crop of zircon, apatite and sphene in a given rock sample, about 1–10 kg of the sample is crushed, depending on the abundance of the materials sought for dating and the size of the available sample. The crushing is carried out in two stages. First the rock sample is crushed by a rock trimmer to reduce it to fragments of 3–5 cm in dimension. Then, the sample is further crushed with a Jaw-crusher to get its powder. To obtain satisfactory yields of the minerals, this stage is repeated a few, say three, times.

2.2. SIEVING

The crushed rock powder is gently washed with water and then sieved through 60 mesh. The powder under 60 mesh is panned with water to remove clay and light minerals. While panning, the water flow is kept very low. After panning, the powder is again sieved through a 200-mesh cloth sieve. During sieving, water is continuously poured in the sieve. The grains between 60–200 mesh are dried in an electric oven for about 12 hours. The temperature of the oven is kept about 60°C so as to avoid the annealing of spontaneous tracks even in the most thermally sensitive mineral, apatite. Before switching this process to the next sample, the 60-mesh sieve is thoroughly washed in an ultrasonic cleaner and the cloth of 200-mesh sieve is changed to new one. The remaining powder over 60 mesh is also dried and stored for a second use, if necessary.

2.3. MAGNETIC AND HEAVY LIQUID SEPARATION TECHNIQUES

The dried rock powder of each sample is processed using isodynamic magnetic separator and heavy liquids. Although this separation process is a standard one, we describe some of its salient features.

After separating the ferromagnetic minerals with a permanent magnet, the sample powder is passed through the isodynamic magnetic separator at 10° forward slope and 0.6 amp current.

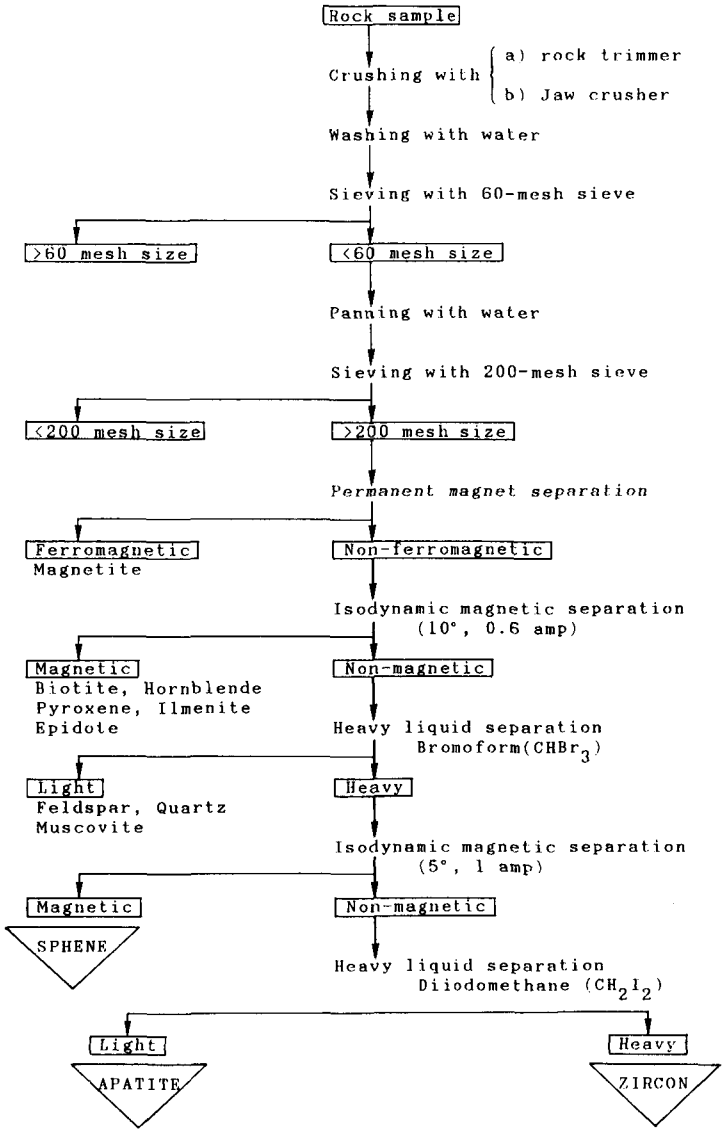

Fig. 1. Flow chart for mineral separation.

(The side slope is always kept at 10°). The magnetic fraction, containing such minerals as biotite, hornblende and pyroxene, is collected in the white aluminum container hanged on the left side; whereas the desirable non-magnetic fraction is collected in the black container on the right.

Before using the magnetic separator, it should be thoroughly cleaned to avoid the contamination. The air compressor with a nozzle is the best device for cleaning. After using

the air compressor for cleaning, we have checked funnel and beakers under microscope and found them free of any contamination.

The non-magnetic fraction that contains the desirable minerals, is then subjected to heavy liquid separation technique. The powder and the bromoform are mixed in a beaker in the ratio of 1:4 by volume (before using the heavy liquid, its density, 2.87 g/cm^3, is checked by standard density glass cubes). In this way, the heavier minerals, zircon, apatite and sphene, sink at the bottom of the beaker, while the lighter minerals, like feldspar and quartz, float in the bromoform which is to be poured into the funnel containing a filter paper. The lighter materials are collected in the filter paper and the bromoform is recovered in the bottle which is kept under the funnel. During the pouring, some lighter materials remain in the beaker as sticking on its walls. These are taken down in the beaker by pouring bromoform with dropper (which should be changed for every new sample) along the sides of the beaker. Bromoform separation is repeated several, say 4–5, times depending upon each sample's conditions, until all lighter materials are separated.

After this separation, the heavy fraction in the beaker is washed thoroughly with alcohol using ultrasonic cleaner, then labeled and kept in an electric oven for a few hours at 60°C for drying. The mixture of bromoform and alcohol is poured in a separation flask of suitable size and mixed with distilled water. This arrangement is kept undisturbed for about 12 hours so that the water absorbs the alcohol and thus bromoform is collected at the bottom of the flask.

The heavy fraction is again passed through the magnetic separator at 5° forward slope and 1 amp current. The magnetic fraction is stored for extracting sphene grains whereas the non-magnetic fraction containing apatite and zircon is again taken through the process of heavy liquid separation using diiodomethane whose density (3.33 g/cm^3) should be checked before using it. During diiodomethane separation, the washing of both the fractions; i.e., light one in the filter paper and heavy one in the beaker, is done with acetone. Diiodomethane is easily recovered by storing the mixture for a long time so that acetone evaporates into the air. Both the fractions are labeled and dried in an electric oven at 60°C for about 1–2 hours. The light fraction contains apatite whereas the heavy, zircon. We find that butter paper is quite convenient for storing the sample fractions.

3. Handpicking and Mounting

Since EDM requires the counting of spontaneous tracks in minerals and that of induced tracks in external detector muscovite, mineral grains and corresponding replica in detector should be identified exactly. For this purpose, some arrangement of mineral grains is desired. Another reason of this grain arrangement is derived from pre-grinding and description procedure, which will be discussed in section 4. In this method, 25 mineral grains in an array of 5×5 or 100 grains in an array of 10×10 grains are arranged and mounted as mentioned below. Three minerals, i.e. zircon, apatite and sphene, are mounted into different materials because they have different track etching and fading conditions. Hence, their handpicking and mounting procedures are described separately.

〈ZIRCON〉

Small fraction of the zircon grains is spread on a silica glass slide of size about 5×5 cm^2. Silica glass is used because the slide is to be heated at 315°C and ordinary glass cracks easily at such a high temperature. In order to avoid the jumping of the grains on the glass slide during spreading, a small quantity of alcohol is put on the glass slide before pouring the zircon grains on it. The glass slide is brought under a stereo biocular microscope having sufficient magnification. Using a thin paintbrush, the grains are picked one by one and arranged at a small place on the glass slide in such a manner that c-axis of all the grains is in one direction (Fig. 2).

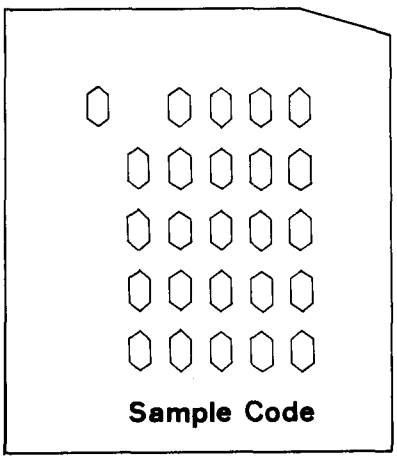

Fig. 2. Zircon grains arranged in an array of 5×5, and mounted in PFA teflon sheet.

In selecting the zircon grains care should be taken that large transparent euhedral crystals having well-defined c-axes, and crystals of approximately same size for a single mount are to be handpicked. One of the corner grains is slightly displaced from the regular array as shown in Fig. 2. The necessity of arranging the grains along their c-axes and the slight displacement of one grain will become clear during the discussion on grinding/polishing and etching of zircon, and track counting in mica detector, respectively. Then, all the remaining grains are removed from the glass slide with the help of a dry paintbrush onto a butter paper and stored after proper labeling.

The silica glass slide is then shifted onto a hot plate having good temperature control. For mounting zircon grains, teflon sheet is generally used because of its stability against the etchant of zircon. FEP (polyfluoroethylene) type teflon (GLEADOW et al., 1976) is used by many workers for this purpose, whose mounting temperature is about 280°C. But is was experienced by one of the authors (TT) that during the etching (especially the prolonged etching up to around 50 hours) the grains in this teflon sheet often got dislodged, and thus an alternative teflon, PFA (copolymer of tetrafluoroethylene-perfluoroalkoxyethylene), was found. This new type of teflon is free from the limitation of FEP because it softens at higher temperature around 320°C. It has been experienced that even with a prolonged etching of 100 hours, grains do not

dislodge from PFA teflon.

In our routine, a sheet of PFA teflon of the size 1.5×1.5 cm^2 and of thickness 0.50 mm is used. Although, the larger thickness is better for keeping the mount flat, the transparency decreases with thickness which, in turn, causes inconvenience in counting tracks under a transmitted light microscope. This teflon sheet is held vertically, using tweezers, on the silica glass slide having zircon grains and heated for about 30 seconds until its bottom end which touches the glass slide melts slightly so as to stay vertically by itself. The placement of the teflon sheet is chosen in such a manner that when tilted slightly with tweezers to allow to fall gently on the glass slide, the zircon grains are mounted in the central area. A second glass plate smaller in size, pre-heated on the same hot plate, is put on the teflon sheet and pressed gently so that the grains get fixed in the teflon sheet. The entire arrangement is then removed from the hot plate and kept on a metallic plate. It is allowed to cool to the room temperature with a small load on the upper smaller glass slide so as not to produce any curvature of PFA sheet during cooling.

The teflon mount, after being removed from the glass slide by applying a little alcohol, is then slightly cut asymmetrically at its right top corner (Fig. 2). This asymmetric cutting makes it convenient to recognize the grain side of the teflon sheet by naked eyes and without using a microscope. The sample code is written with a needle pen on the backside of the teflon mount.

〈APATITE〉

Since fission tracks in apatite are very sensitive to thermal effect, its mounting in teflon like that of zircon should be avoided. Instead, an alternative procedure that does not involve high temperature treatment is employed. Normally, epoxy resin is used for the mounting of apatite grains, where adhesive and hardener are mixed in the ratio 2 : 1.

The epoxy resin mount after drying cannot be detached from the glass slide but can easily be removed from the teflon slide, which is, therefore, very useful for apatite mounting. The teflon slide is attached on the glass slide of the same size using bifacial tape (henceforth this combination of glass and teflon slide will be called TG slide). TG slide is cleaned with alcohol and a little vaseline is applied on the teflon side of the slide so that the apatite grains can stick to it. Unless, the electrostatic effect will disturb the grain arrangement during the hand-picking.

A small quantity of apatite grains is spread on an ordinary glass slide as it is done in the case of zircon grains handpicking. For convenience during the handpicking, this glass slide should have the same thickness as that of the TG slide. The glass slide containing apatite grains and the TG slide are put adjacent to one another under a stereo binocular microscope. The handpicking of apatite grains is done similar to that of zircon, but no need to be aligned in one direction.

All the TG slides containing apatite grains are arranged on a horizontal surface. On each slide, two small glass pieces of 1.5 mm thick are placed as spacers. Then the epoxy resin of fixed amount is poured on the grains carefully so that the grain arrangement is not disturbed.

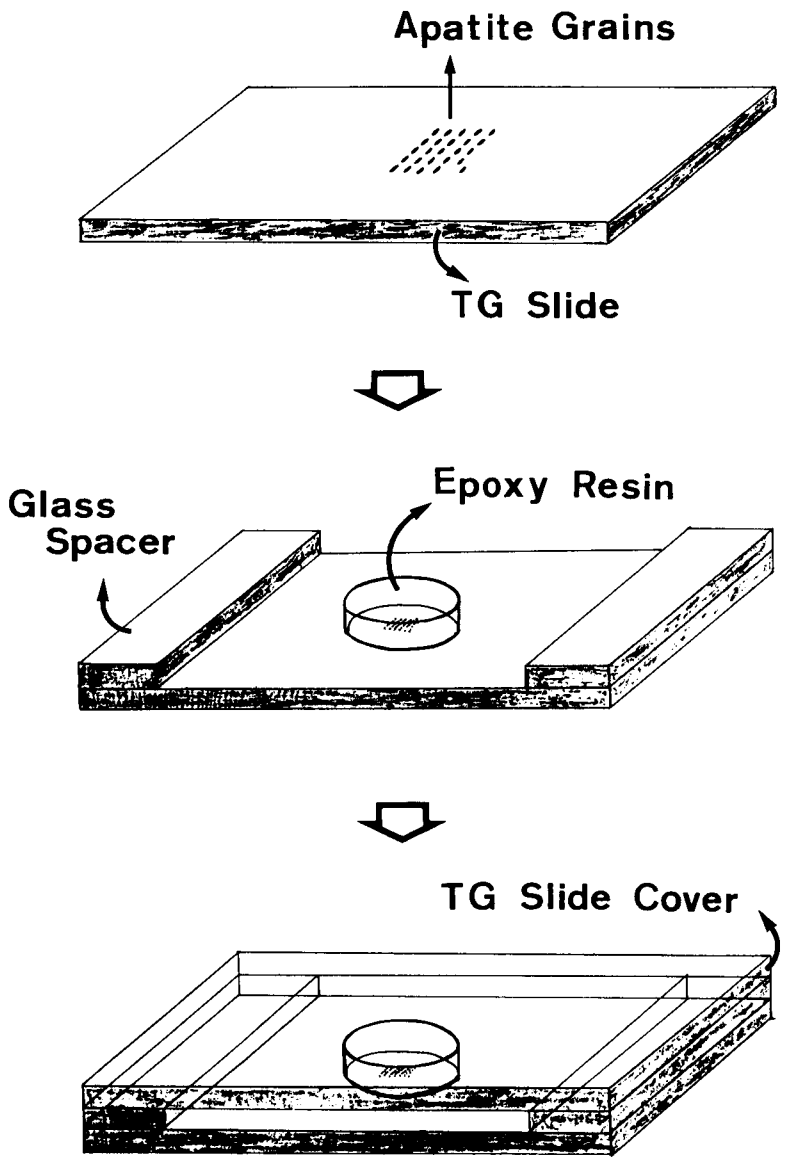

Fig. 3. Mounting procedure for apatite grains in epoxy resin.

The amount of the epoxy resin is adjusted in such a manner that when another TG slide is put (with teflon on the lower side) on the glass spacers, the poured resin makes a flattened cylindrical form whose diameter is about 15 mm. The entire process is illustrated in Fig. 3. The quantity of epoxy resin can be measured with a disposable plastic syringe.

This arrangement is kept for about 24 hours at room temperature to allow the epoxy resin

to solidify. Slight heating, which should be kept below 100°C, will accelerate the solidification. The mount is removed easily due to the flexibility of the teflon slide. The sample code is written with a needle pen parallel to one of the four sides of the grain arrangement on the backside of the mount.

〈SPHENE〉

The handpicking and mounting of sphene grains can be carried out in any of the two ways as described for zircon and apatite. In the case of sphene, the crystal alignment need not be taken into consideration. When using teflon, FEP is preferred to PFA, since it is easily flattened during cooling after the mounting.

4. Grinding and Polishing

For FTD of uranium bearing minerals, such as zircon, apatite and sphene, internal crystal surface is generally used for measuring spontaneous track density. Flat internal surfaces of the mineral grains are exposed by grinding and polishing procedure. Due to small thickness of the mineral mount, it becomes inconvenient to hold it during the grinding/polishing. To overcome this problem, the mount is fixed on a transparent acrylic block of suitable size ($5 \times 4 \times 1$ cm^3) using bifacial tape. Although the sample code is written on the back of the mount, during the grinding/polishing it may be difficult to read it through the acrylic block and hence the sample code is also written on a strip of scotch magic tape with magic pen and stuck on the acrylic block. Another piece of the magic tape is also stuck to cover the sample code and to avoid its probable disappearance.

For using internal surfaces of the minerals, it is essential to remove a certain thickness to expose 4π geometry. The thickness corresponds to half of etchable track length, which varies with each mineral; $\sim 6\,\mu$m for zircon (KRISHNASWAMI et al., 1974); $\sim 8\,\mu$m for apatite (GLEADOW et al., 1986); $\sim 7\,\mu$m for sphene (GLEADOW and LOVERING, 1977). If all the mineral grains are mounted such that the exposed crystal surface is equally flat, then the grains will be ground almost uniformly and the removal of the thickness which satisfies 4π geometry will be quite easy. However, in case mineral grains are inclined, eroded, or have irregular shape or caves, all the surface area of each grain will not be ground equally and hence the measurement of the removed thickness for 4π geometry condition will not be possible from the first grinding. In such case, before measuring the thickness, grinding should be continued till maximum possible area is exposed (henceforth, this step will be called as "pregrinding"). The pre-grinding is carried out in the following manner.

The mount is ground gently in one direction several times on the emery paper of 1500 mesh size (wetted with water) pasted on a thick glass plate, and observed under microscope for the grinding scratches. If the grinding scratches are observable on all the grains, pre-grinding is complete. Otherwise, it is continued until the maximum possible area is scratched.

In case the size of the grains on the same mount is quite different from each other, it is possible that due to continued grinding (to make scratches appear on all grains) the smaller

grains may become so thin that with further grinding they may get detached from the teflon sheet. This necessitates to have grains of almost same size for a mount (that should be taken into consideration during the handpicking).

After pre-grinding, a brief description about the suitability of each grain is necessary. As discussed above, during pre-grinding some part of the crystal grain may not be exposed and not satisfy the condition for 4π geometry. As it is impossible to judge such non-exposed area after finishing the grinding/polishing, all these areas are recorded at this stage in a notebook by labeling individual grains.

The next step is to grind the mount and remove a certain thickness peculiar to each mineral for exposing 4π geometry. The removed thickness can be measured by choosing some surface feature, such as exposed intrusion, on a grain and noting the removal of surface feature depth by z-motion of microscope. In our experience, the depth of the grinding scratch is found to be at least 1 μm using wetted emery paper of 1500 mesh. This knowledge serves us simpler way of measuring the removed thickness during grinding, as mentioned below.

After the description, the mount is ground perpendicular to the direction of the pre-grinding till all the preceding grinding scratches disappear. This ensures that all the exposed surface area has been ground and removed for at least 1 μm. Next step of grinding is carried out again perpendicular to the direction of the last grinding and new scratches of all surface area are checked, confirming the removal of another 1 μm. This process is repeated until the removed thickness reaches the half of etchable track length for each mineral. For safety, however, the thickness of the removed surface is kept more than 3/2 of the minimum depth for exposing 4π geometry. Thus each step of grinding is repeated, in our routine, 9 times for zircon, 12 times for apatite and 11 times for sphene.

In case of zircon, the grinding procedure is slightly different from those of the other two minerals. It is not preferred to grind zircon crystals along their crystallographic c-axes because it often produces, in our experience, deep cracks or damages on the surface. The cracks or damages are enlarged during chemical etching and sometimes disturb the track counting. Hence, the pre-grinding and grinding of zircon mounts should be carried out only perpendicular to c-axis, for which the arrangement of zircon crystals along c-axis is desired. After each step of grinding, all the grinding scratches are erased by 7 μm diamond paste polishing parallel to c-axis, so that next grinding scratches can be easily observed.

After the grinding, the mount is polished successively with 7 μm, 2.5 μm, 1 μm and 0.25 μm diamond paste. At every step of polishing, the direction is changed by 90° from that of preceding polishing. The alternately changing of polishing direction makes it easy to distinguish the disappearance of the preceding polishing scratches, ensuring thereby the completion of each polishing step. Since it may be difficult to check the removal of the scratches with transmitted light microscope, reflected light microscope is very useful for this purpose. Before switching from one diamond paste to another, the samples are washed with running water and ultrasonic cleaner to make them clean of the preceding diamond paste. For all the three minerals, polishing time with each diamond paste should always be about five times larger than that just required to erase the scratches of the preceding paste.

After finishing grinding and polishing, the mount is removed from the acrylic block and the sample is ready for etching.

5. Etching of Fission Tracks in Minerals

Fission tracks in a mineral grain can be enlarged and made visible in an optical microscope by chemical etching. Underetched tracks are faint and difficult to observe under a microscope so that the track density is generally underestimated. On the other hand, overetched tracks are difficult to discriminate reliably from other etched features and because of their size, may overlap each other if track density is high. Ideally, therefore, tracks should be etched to somewhere between these two extremes for reliable track identification and counting. In order to determine this optimum etching time, progressive etching and counting experiment is performed for a given mineral and the time corresponding to the track density saturation gives the optimum etching time. We routinely carry out this experiment for pilot mount of zircon, apatite and sphene samples of unknown age because their optimum etchsing time is known to vary from sample to sample.

Especially for zircon and sphene, we have to be very judicious in etching. As demonstrated by GLEADOW (1978, 1981) and SUMII et al. (1987), these two minerals show anisotropic etching characteristic of fission tracks and all the tracks in various crystallographic direction do not have same etching rate. Therefore it is necessary to continue the etching until the tracks in all direction become visible. In case of zircon, isotropic distribution of etched tracks is characterized by the complete revelation of thin tracks parallel to c-axis.

The optimum etching time of zircon and sphene can vary, even in one sample, from grain to grain and area to area due to large variation of spontaneous track density. It is quite possible that some of the grains are optimally etched whereas others may require more etching for the appearance of tracks in a certain direction of low track etching rate. In such case, the decision for further etching can be taken on the basis of the number of grains which are underetched. If the observer feels that further etching makes more grains available for dating than the number it spoils by overetching, he can etch the sample further.

Etching procedures for the three minerals are different, and hence will be mentioned separately.

〈ZIRCON〉

For etching a large number of zircon mounts together, an electric hot plate having holes for keeping teflon beakers is used (Fig. 4). The depth of these holes can be made slightly smaller than the height of the teflon beaker containing the eutectic NaOH : KOH (1 : 1) etchant (GLEADOW et al., 1976). The temperature of this heater is controlled with the help of a thermocouple and a temperature controller. The constancy of the temperature with time can be monitored by connecting the output of the thermocouple to x-t recorder. In order to get the difference between the temperature of the heater and the etchant, another similar thermocouple is dipped in the etchant inside the beaker and its output is fed to the x-t recorder.

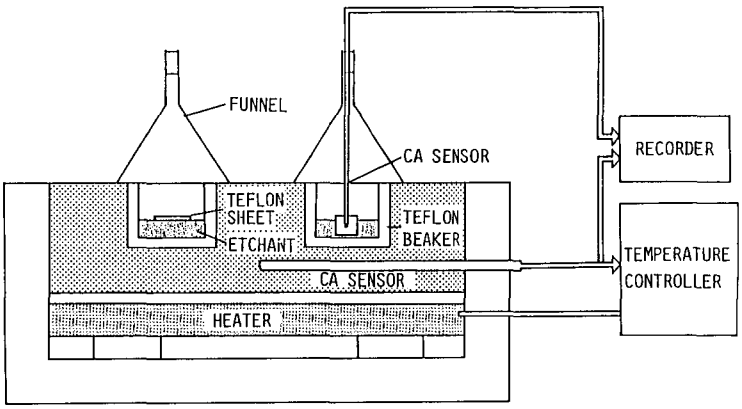

Fig. 4. Etching system for zircon using eutectic NaOH : KOH.

After the required temperature of the heater ($225\pm0.5^{\circ}C$) is attained, the etchant is put into the beakers (the number of beakers can be selected according to the number of samples and the space available in the etching heater) and the beakers are kept in the heater for about 12 hours and covered with funnels. Within this period, no air bubbles are left in the etchant.

Then 4–5 zircon mounts are put in each beaker for etching. The etching time of Fish Canyon Tuff zircon standard ($\rho_s\sim5\times10^6\ cm^{-2}$) is about 25 hours in our system. After taking out the zircon mount from the etchant, it is put in 5% HCl in an ultrasonic cleaner for about 15–20 minutes to remove the traces of the etchant sticking to the mount. We have found from our experience that the etchant once prepared can be used for one week and then should be replaced by new one.

It sometimes happens during etching that the PFA teflon sheet slightly loses its flatness probably due to the long, high temperature treatment. Such a curvature makes the microscopic observation somewhat difficult, and will disturb the complete attachment of the muscovite detector during irradiation. To resolve this problem, we sandwich the teflon sheet between two silica glass slides and put them, with a small weight, in an electric oven at 250°C for a few hours, and the mount will become flat.

〈Apatite〉

We etch apatites in 0.6% HNO_3 at $32.0\pm0.5^{\circ}C$ in a constant-temperature water bath. In this system, it takes about 60 sec. to etch Fish Canyon Tuff apatite standard ($\rho_s\sim2\times10^5\ cm^{-2}$).

〈Sphene〉

Etching of sphene is commonly done in any of the two ways: a) using NaOH at 130°C (Calk and Naeser, 1973), b) using the acid mixture; $HF:HNO^3:HCL:H_2O=1:2:3:6$ at room temperature (Naeser and McKee, 1970). No significant difference of etched track shapes and resulting ages between these two methods have been reported and both have been used successfully by many workers. We, however, prefer the latter acid mixture etchant

because with which the etching efficiency of sphene is well studied (GLEADOW, 1978) and also experimental treatment at room temperature is easier. The temperature of the etchant is maintained at 32.0 ± 0.5°C and we have found that it takes about 6 minutes for Mt. Dromedary sphene standard ($\rho_s\sim5\times10^6$) to be etched.

After etching is finished, all the mineral mounts are thoroughly washed with distilled water using an ultrasonic cleaner and dried under an infra-red lamp for about an hour.

6. Packing and Irradiation

〈PACKING〉

In EDM, fission track detector is fixed against the etched mineral surface during the thermal neutron irradiation. The uranium free muscovite sheet, such as Brazilian muscovite, is routinely used as a detector. It is cut into square pieces of size slightly larger than the area of the mounted grains and of suitable thickness (~0.1 mm).

In order to get reliable data, the external detector should be free of any external contamination. For getting a contamination-free and freshly cleaved surface of muscovite, one end of a small strip (~5 cm) of scotch magic tape is stuck to the edge of a lab. table and, then, using tweezers the trimmed muscovite is stuck on it. Now a jerk is given with hand such that the muscovite is cleaved off from the lower flake sticking to the tape. This process may be repeated 2–3 times until we get an even fresh internal surface of muscovite.

The sample code is written with a needle pen on the back of the muscovite. To distinguish the surface fixed against mineral grains, one corner of it is cut asymmetrically such that when fixed against the grains arrangement in the teflon sheet, the cuts of both are in the

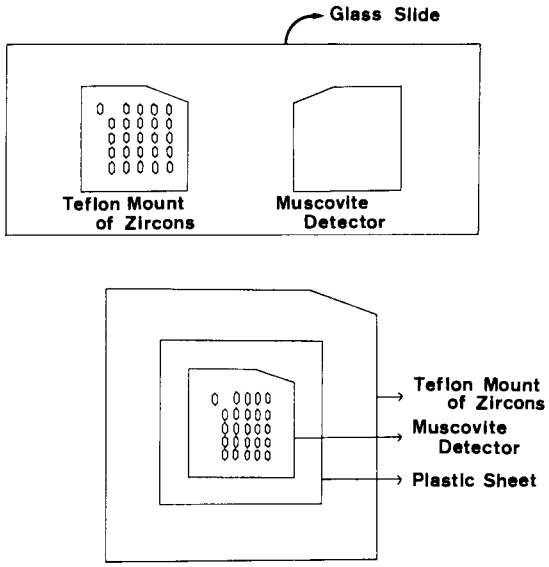

Fig. 5. Packing of zircon mounts for thermal neutron irradiation.

same dirdection (Fig. 5). For epoxy-resin mounts, the corner cut of muscovite detector is made in the same manner.

Before putting detectors, all the mineral mounts are observed under microscope to see if there is any undesirable particles, such as dust, sticking to the mount. If so, they are handpicked with the paintbrush.

After keeping the mica detector on the grains, it is covered with a clean plastic sheet (~0.5 mm) of size slightly larger than the muscovite (plastic sheet used to avoid the sticking of this tape to the mica detector). The entire arrangement is, then, wrapped up firmly with scotch magic tape and the sample code is written on the tape. The sample is now ready for irradiation.

〈Thermal Neutron Irradiation〉

After fixing the mica detectors on mineral mounts, samples are stacked vertically and packed in a capsule, the size of which depends on the irradiation facility in the reactor. In order to measure thermal neutron fluence and to take into account dose gradient along the capsule, two uranium standard glasses (one on the top and another at the bottom) are also packed along with the samples. The type of the uranium standard glass is decided by the thermal neutron fluence to which the samples are to be exposed (e.g. if the dose is high, low uranium glass is used).

In our case we use the Pneumatic tube facility of Kyoto University Research Reactor (KUR-1) and Irradiation pit facility of Musashi Institute of Technology Reactor (MITR). In KUR-1, the thermal neutron flux is 2.8×10^{13} n/cm^2/sec and the Cd ratio in Au is 5.8. The corresponding valsues in MITR are 8.0×10^{11} n/cm^2/sec and 13–15. The dosimeter glass mounts used for irradiation in KUR-1 and MITR consist of three glasses: CN1, CN2 and NBS-SRM612, SRM962a, the details of which are given in Table 1. They are mounted similar to that of apatite grains. The muscovite detectors attached to this mount are used for counting induced fission tracks produced during the irradiation.

Table 1. Description of Uranium Standard Glasses

Glasses	Uranium content (ppm)	^{235}U isotopic abundance (%)
CN*1	~39	0.726
CN2	~36	0.726
NBS**-SRM612, 962a	37.38±0.08	0.2392

* Corning
** National Bureau of Standards

7. Sample Analysis and Data Collection

7.1. Etching of Muscovite Detector

After thermal neutron irradiation of samples, muscovite detectors are removed from the

mounts. First the tape and the plastic is removed, and then, the mount is put in alcohol for 2–3 minutes. The muscovite detector is easily detached from the mount in this way.

A special arrangement is made so as to etch a large number of muscovite detectors simultaneously. A rubber ring which is highly resistant to HF is taken. About 10–12 small cuts are made on the ring of inner diameter 30 mm and the thickness 3.5 mm with a sharp blade. With the help of tweezers, one muscovite detector is fixed in each of these cuts in such a manner that only its one side goes inside the cut (Fig. 6). The muscovite will get fixed in the ring due to the rubber ring's elasticity. These rubber rings can be repeatedly used until the cuts become so wide as not to be able to withhold the muscovite detectors.

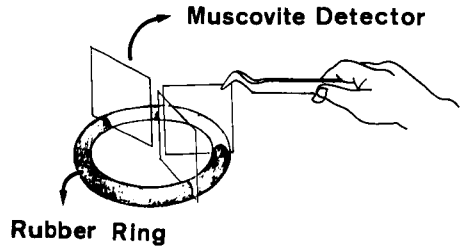

Fig. 6. Fixing of muscovite detectors in a rubber ring.

The ring containing the muscovites is put in a plastic beaker having some holes on its walls. For starting the etching, this beaker is put into 47% HF in a constant-temperature water bath at 32.0 ± 0.5°C. The optimum etching time for muscovite detectors in this etching condition is found to be 4–5 minutes by the preliminary progressive etching.

After etching, the plastic beaker containing the muscovites is immersed in distilled water in a glass beaker and put in an ultrasonic cleaner for a few minutes. The plastic beaker is then dried under an infra-red lamp for about an hour to evaporate HF vapors perfectly.

The muscovite detectors and the sample mounts are fixed in the following manner: on one side of a glass slide, a paper sticker is fixed on which the sample code is written. The sample mount and its corresponding mica detector are also fixed on the glass slide using transparent manicure (Fig. 7). The manicure is used because it easily dries and is soluble in organic substances like benzene and alcohol, and hence the sample or muscovite detector can be removed from the glass slide if necessary. The mineral mount and its mica replica must be fixed in such a position that the uppermost line of grains in both the mount and replica hold almost the same horizontal position to make the track counting convenient. The sample and its corresponding mica detector is now ready for the next stage—counting of fission tracks.

7.2. COUNTING OF TRACKS

Etched fission tracks on the mineral grains (zircon, apatite and sphene) and muscovite detectors are counted using Nikon Biophot microscopic system with 100× dry objective. Dry objective is preferred because it is possible to miss shallow tracks with oil immersion objectives,

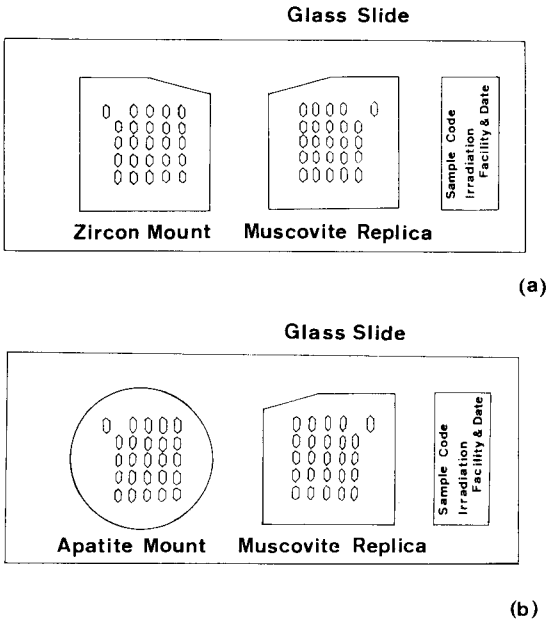

Fig. 7. Fixing of irradiated mineral mount and corresponding muscovite replica on a glass slide for track counting.
(a) for zircon. (b) for apatite.

especially in case of apatite and muscovite. The overall calibrated magnification is 925× in our system.

While counting the tracks in any of the mineral grain, first, shape of the grain is drawn on the paper and suitable area for counting is chosen according to the description of each grain (see section 4). The counted area is also marked with the help of a graticule fitted in one of the eye-pieces of the microscope. After counting the tracks in the mineral grain, the corresponding area in the muscovite replica of the grain is also scanned for counting the induced tracks. The scanning is quite easy in our system because mineral grains and corresponding muscovite replica are arranged in an array of 5×5 or 10×10 crystals.

Number of tracks to be counted depends on the statistical precision required for the age determination. The criteria for discriminating fission tracks from other etched features are well discussed in Fleischer and Price (1964). In case of zircon and sphene, only the grain surfaces of high etching efficiency which are easily judged by the existence of sharp polishing scratches (GLEADOW, 1981), and the optimally etched grains or areas which show isotropic angular distribution of etched tracks should be used for counting.

For the thermal neutron fluence measurement, the muscovite replica of the dosimeter glass is uniformly scanned with help of a scale attached on the mechanical stage of the microscope. About 1000 tracks are totally counted for each dosimeter glass.

7.3. AGE AND ERROR CALCULATIONS

The FT age is calculated by using the formula

$$T=\frac{1}{\lambda_D} ln\left[1+\frac{\lambda_D \sigma \Phi I g \rho_s}{\lambda_f \rho_i}\right] \quad \cdots\cdots(1)$$

where λ_D is the total decay constant of ^{238}U; λ_f is the spontaneous fission decay constant of ^{238}U; σ is the thermal neutron fission cross-section for ^{235}U; Φ is the thermal neutron flux; I is the isotopic abundance ratio $^{235}U/^{238}U$; g is a geometry factor; ρ_s is the spontaneous fission track density of the sample; and ρ_i is the induced fission track density of the sample.

Besides the measurement of ρ_s and ρ_i, we need to estimate Φ for age calculation. Φ can be measured with help of the track density (ρ_d) in muscovite detector attached to the dosimeter glass durisng the irradiation, using the relationship

$$\Phi = B\rho_d \quad \cdots\cdots(2)$$

where B is a constant which can be determined experimentally against Au, Cu, Co, or uranium thin source flux monitors. This was the usual practice to calculate until HURFORD and GREEN (1981, 1982) pointed out the possibility of some systematic errors in determination of B. Besides, the selection of a unique value for λ_f was also a problem as there is wide range of λ_f values repeated in the literatures (see THIEL and HERR, 1976; BIGAZZI, 1981). In order to overcome the problem of λ_f and B, HURFORD and GREEN (1982, 1983) proposed an alternative way of calibration using a age standards, which is named zeta calibration.

In zeta calibration method, a dosimeter glass is calibrated against an age standard, and then the calibrated factor zeta is applied to age determination of unknown samples. The unknown age (*Tunk*) can be calculated by rewriting Equation (1) and (2) with factor zeta (ζ) replacing λ_f, σ, I and B. Thus,

$$Tunk=\frac{1}{\lambda_D} ln\left[1+\zeta \lambda_D g \frac{\rho_s}{\rho_i}\rho_d\right] \quad \cdots\cdots(3)$$

where ζ for the glass is evaluated from an age standard according to

$$\zeta=\frac{(e^{\lambda d Tstd}-1)}{\lambda_D g\left[\frac{\rho_s}{\rho_i}\right]_{std}\rho_d} \quad \cdots\cdots(4)$$

where *Tstd* is the reference age of the standard.

The statistical error (σ_T) associated with the FT age can be calculated by using the formula

$$\sigma_T = T\sqrt{\frac{1}{N_s}+\frac{1}{N_i}+\frac{1}{N_d}+\left(\frac{\sigma\zeta}{\zeta}\right)^2} \quad \cdots\cdots(5)$$

where N_s is the counted number of spontaneous tracks in the sample; N_i is the counted number

of induced tracks in the sample; N_d is the counted number of induced tracks in the dosimeter glass; and σ_ζ is the statistical error of ζ and is given by

$$\sigma_\zeta=\zeta\sqrt{\left(\frac{\sigma_{Tstd}}{T_{std}}\right)^2+\left[\frac{1}{N_s}+\frac{1}{N_i}\right]_{std}+\frac{1}{N_d}} \qquad \cdots\cdots(6)$$

where σ_{Tstd} is the error of the reference age of the standard.

In order to check the correlation of spontaneous and induced track densities in various grains of a sample, the correlation coefficient (*c.c.*) can be evaluated by using the formula

$$c.c.=\frac{\sum_{k=1}^{N}(\rho_i(k)-X_i)\cdot(\rho_s(k)-X_s)}{\sqrt{\sum_{k=1}^{N}(\rho_i(k)-X_i)^2\cdot\sum_{k=1}^{N}(\rho_s(k)-X_s)^2}} \qquad \cdots\cdots(7)$$

where

$$X_i=\frac{\sum_{k=1}^{N}\rho_i(k)}{N} \text{ and } X_s=\frac{\sum_{k=1}^{N}\rho_s(k)}{N} \qquad \cdots\cdots(8)$$

7.4. Computer Program

In order to obtain the table of analytical data, the $\rho_s-\rho_i$ diagram and the histogram of grain data for calculating and examining ζ value and FT age, a computer program is given as an appendix which can be run on most of the personal computers. For multiple determination of ζ value for each standard and FT age for each sample, the error component from the reference age of the standard and that from the zeta constant are not contained in resulted zeta value and FT age, respectively.

The input data are as follows:

Sample code, N_s, N_i, counted area of spontaneous and induced tracks (A), name of dosimeter glass, N_d, counted area of dosimeter glass (A_d), *Tstd.* or ζ.

Of these data, sample code, N_s, N_i and A are to be input separately on another disk (drive *B*) using Word Star.

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Appendix

```
1000 REM ###  PROGRAM OF FISSION TRACK DATING  ###
1010 CLS 3
1020 LOCATE 0,2:PRINT "  PROGRAM OF FISSION TRACK DATING "
1030 LOCATE 0,4:PRINT "  NOTICE : When you print out 'age table', 'ze
ta table' and 'list of input data',"
1040 LOCATE 0,5:PRINT "  please use 'EPSON UP-130K(PC) printer'."
1050 LOCATE 0,7:PRINT "  When you print out 'age histogram','zeta his
togram','log ρs- log ρi diagram'"
1060 LOCATE 0,8:PRINT "  and 'ρs - ρi diagram', please use 'NEC PC-8
826 plotter'."
1070 PRINT
1080 PRINT :PRINT :INPUT " -- Push return key to start.",A
1090 '
1100 CLS 3:LOCATE 10,4:PRINT "  Data should be input as below in anot
her disk "
1110 LOCATE 10,5:PRINT "  by using WordStar."
1120 LOCATE 10,10: PRINT "Fish Canyon Tuff     -------  Sample code
"
1130 LOCATE 10,11:PRINT "150 122 30           -------  Ns Ni A "
1140 LOCATE 10,12:PRINT "180 200 20           -------  Ns Ni A "
1150 LOCATE 10,13:PRINT "170 110 25           -------  Ns Ni A "
1160 LOCATE 0,20:INPUT "-- Push return key to start.",A
1170 '
1180 CLS 3:LOCATE 5,5:INPUT" You want  1) zeta calibration   2) age c
alculation ? ",XYZ
1190 IF XYZ=1 THEN 6140
1200 CLS 3
1210 INPUT "    sample code  (B:file name)  ";S$ :PRINT
1220 INPUT " Your sample ? ---- 1) zircon  2) apatite  3) sphene  ",S
M:PRINT
1230 IF SM=1 THEN SM$="zircon"
1240 IF SM=2 THEN SM$="apatite"
1250 IF SM=3 THEN SM$="sphene"
1260 REM ### data of spontaneous tracks ###
1270 CLS 3
1280 PRINT "--- For spontaneous tracks ---"
1290 PRINT
1300 PRINT "On which surface did you count spontaneous tracks,"
1310 INPUT "    1) internal surface (ED1) or  2) external surface (ED
2) ? ",QS :PRINT
1320 DIM F(900)
1330 OPEN S$ FOR INPUT AS #1
1340 INPUT #1,A$
1350 I=O
1360 I=I+1
1370 INPUT #1,F(I)
1380 IF EOF(1) THEN 1400
1390 GOTO 1360
1400 CLOSE #1
1410 S$=MID$(S$,3)
1420 N=I/3
1430 DIM AA(I),BB(I),CC(I),DD(I)
1440 DIM A(N),B(N),PS(N),C(N),D(N),PI(N),T(N),E(N),CL(100),CA(100),Z(
N),EZ(N)
1450 DIM PIC(N),PSD(N),PIA(N),PIB(N),PID(N),PIE(N),PSA(N),PSB(N)
```

```
1460 DIM PSE(N),PSF(N),X(N),Y(N),GS(N),GI(N)
1470 FOR I=1 TO N
1480 J=-2+3*I:K=-1+3*I:L=3*I
1490 AA(J)=F(J):BB(L)=F(L):CC(K)=F(K):DD(L)=F(L)
1500 A(I)=AA(J):B(I)=BB(L):C(I)=CC(K):D(I)=DD(L)
1510 NEXT I
1520 PRINT
1530 INPUT "1 grid area = 1.162x10^(-6) cm^2  (y or n)";A$
1540 PRINT
1550 AG=1.162
1560 IF A$="n" THEN INPUT "1 grid area =    x10^(-6) cm^2  ";AG
1570 PRINT
1580 FOR I=1 TO N
1590 B(I)=B(I)*AG
1600 PS(I)=A(I)/B(I)
1610 NEXT I
1620 REM ### data of induced tracks ###
1630 CLS 3 :PRINT
1640 PRINT
1650 PRINT "--- For induced tracks ---"
1660 PRINT
1670 INPUT "1 grid area = 1.162x10^(-6) cm^2  (y or n)";A$
1680 PRINT
1690 AG=1.162
1700 IF A$="n" THEN INPUT "1 grid area =   x10^(-6) cm^2 ";AG
1710 PRINT
1720 FOR I=1 TO N
1730 D(I)=D(I)*AG
1740 PI(I)=C(I)/D(I)
1750 NEXT I
1760 IF XYZ=1 THEN 6240
1770 REM ### calculation of age ###
1780 CLS 3
1790 PRINT "--- calculation of age ---  "
1800 PRINT
1810 INPUT "  Name of dosimeter glass ?  ",ST$:PRINT
1820 INPUT "  Zeta value ";Z
1830 PRINT
1840 INPUT "  Track number of dosimeter glass ";ND
1850 PRINT
1860 INPUT "  Counted area of dosimeter glass (x100 grids) ";CAS
1870 TD=ND/(CAS*100*AG)
1880 FOR I=1 TO N
1890 T(I)=10^4*LOG(1+1.55125*10^(-4)*Z*PS(I)*TD/PI(I))/1.55125
1900 IF QS=1 THEN T(I)=10^4*LOG(1+1.55125*10^(-4)*Z*PS(I)*TD/(PI(I)*2
))/1.55125
1910 IF A(I)=0 THEN E(I)=T(I)*SQR(1/C(I)+1/ND):GOTO 1930
1920 E(I)=T(I)*SQR(1/A(I)+1/C(I)+1/ND)
1930 NEXT I:CLS 3
1940 PRINT
1950 '  correlation coefficient
1960 PI=0:PS=0
1970 FOR I=1 TO N
1980 PI=PI+PI(I):PS=PS+PS(I)
1990 NEXT I
2000 XI=PI/N:XS=PS/N
2010 R=0:VI=0:VS=0
2020 FOR I=1 TO N
```

```
2030 R=R+(PI(I)-XI)*(PS(I)-XS)
2040 VI=VI+(PI(I)-XI)^2
2050 VS=VS+(PS(I)-XS)^2
2060 NEXT I
2070 CC=R/SQR(VI*VS)
2080 LOCATE 5,2 :PRINT "   Which menu do you need ? "
2090 IF XYZ=1 THEN 6350
2100 LOCATE 5,4 :PRINT "    1) age table   2) age histogram  3)log ρ
s - log ρi diagram  "
2110 LOCATE 5,6:PRINT "   4) copy of '1)'  5) copy of '2)'  6) copy o
f '3)'   "
2120 LOCATE 5,8:PRINT "   7) ρs - ρi diagram   8) copy of '7)'  "
2130 LOCATE 5,10 :PRINT "  9) list of input data   10) copy of '9)'
"
2140 INPUT A :CLS 3
2150 ON A GOTO 2170,2490,2930,3260,3620,4120,4530,5190,5900,6020
2160 END
2170 '
2180 IF XYZ=1 THEN 6370
2190 REM ### Age table ###
2200 PRINT
2210 WIDTH 80,25
2220 PRINT USING"     @  (@)    ζ(@) = ####.#      ρ(@) =##.#### (N =
#####)  ";S$,SM$,ST$,Z,ST$,TD,ND
2230 PRINT STRING$(75,"-")
2240 PRINT "sample    spontaneous tracks";TAB(32);"induced tracks";TA
B(52);"age"
2250 PRINT " no.";TAB(10);"number";TAB(20);"density";TAB(30);"number"
;TAB(40);"density";TAB(53);"Ma  (±1σ) "
2260 PRINT STRING$(75,"-")
2270 FOR I=1 TO N
2280 PRINT USING" ###       #####   ####.####  #####    ####.####  ##
##.### ±###.### ";I,A(I),PS(I),C(I),PI(I),T(I),E(I)
2290 NEXT I
2300 PRINT STRING$(75,"-")
2310 PRINT :PRINT
2320 '
2330 A=0:B=0:C=0:E=0
2340 FOR I=1 TO N
2350 A=A+A(I):B=B+B(I):C=C+C(I):E=E+D(I)
2360 NEXT I
2370 PS=A/B:PI=C/E
2380 T=10^4*LOG(1+1.55125*10^(-4)*Z*PS*TD/PI)/1.55125
2390 IF QS=1 THEN T=10^4*LOG(1+1.55125*10^(-4)*Z*PS*TD/(PI*2))/1.5512
5
2400 F=T*SQR(1/A+1/C+1/ND)
2410 PRINT USING " total    #####    ####.####  #####    ####.####  #
###.### ±###.###";A,PS,C,PI,T,F
2420 PRINT
2430 PRINT USING"  correlation coefficient = #.###";CC
2440 PRINT
2450 IF QS=2 THEN PRINT " spontaneous track surface  : 2π":PRINT
2460 INPUT"  1) continue     2) finish   ";A
2470 IF 2=A THEN END
2480 CLS 3:GOTO 2080
2490 '
2500 REM ###  age or zeta histogram  ###
2510 WIDTH 80,25:CLS 3
```

```
2520 CONSOLE,,,1
2530 SCREEN 0,0
2540 IF XYZ=1 THEN 6660
2550 PRINT "                          age histogram    " :PRINT
2560 INPUT "   Width of age = ";A :CLS 3
2570 LINE (80,160)-(560,0),,B
2580 FOR I=1 TO 19
2590 LINE (80+24*I,160)-(80+24*I,157)
2600 NEXT I
2610 FOR I=1 TO 3
2620 LINE (80+120*I,160)-(80+120*I,154)
2630 NEXT I
2640 FOR I=0 TO 4
2650 LOCATE 8+15*I,21:PRINT 5*I*A
2660 NEXT I
2670 FOR I=1 TO 4
2680 LOCATE 7,20-I*5:PRINT USING "##";5*I
2690 LINE (80,160-I*40)-(85,160-I*40)
2700 NEXT I
2710 IF XYZ=1 THEN 6690
2720 MAX=T(1)
2730 FOR I=2 TO N
2740 IF MAX<T(I) THEN MAX=T(I)
2750 NEXT I
2760 G=INT(MAX/A)+1
2770 FOR I=1 TO G
2780 CL(I)=0
2790 NEXT I
2800 FOR I=1 TO N
2810 F=INT(T(I)/A)+1
2820 CL(F)=CL(F)+1
2830 NEXT I
2840 FOR I=1 TO G
2850 J=I+1
2860 LINE (56+I*24,160)-(54+J*24,160-CL(I)*8),4,BF
2870 NEXT I
2880 LOCATE 10,23:PRINT " 1) continue      2) finish  ?  "
2890 INPUT A
2900 IF 2=A THEN END
2910 CLS 3 :GOTO 2080
2920 '
2930 REM ###  log ρs -log ρi diagram     ###
2940 SCREEN 3:CONSOLE,,0:WIDTH 80,25
2950 CLS 3
2960 FOR I=1 TO N
2970 IF A(I)=0 THEN PRINT " Not suitable for log ρs -log ρi diagram
"
2980 NEXT I
2990 FOR I=1 TO N
3000 IF A(I)=0 THEN GOTO 3220
3010 NEXT I
3020 WINDOW(-.1,-3.6)-(3.6,.1)
3030 VIEW(160,0)-(520,360)
3040 LINE(0,0)-(3.5,0):LINE(0,0)-(0,-3.5)
3050 FOR I=1 TO 3
3060 LINE(I,0)-(I,-.05):LINE(0,-I)-(.05,-I)
3070 NEXT I
3080 FOR I=1 TO 3
```

```
3090 J=19+13*I:K=4+I
3100 LOCATE J,22:PRINT K
3110 NEXT I
3120 FOR I=1 TO 3
3130 J=21-6*I:L=4+I
3140 LOCATE 18,J:PRINT L
3150 NEXT I
3160 FOR I=1 TO N
3170 X(I)=LOG(PI(I)*10^6)/LOG(10)-4
3180 Y(I)=-LOG(PS(I)*10^6)/LOG(10)+4
3190 CIRCLE(X(I),Y(I)),.03
3200 NEXT I
3210 PRINT :PRINT USING"   cc = #.###";CC
3220 LOCATE 10,23:PRINT " 1) continue     2) finish ?  "
3230 INPUT A
3240 IF 2=A THEN END
3250 CLS 3 :GOTO 2080
3260 '
3270 IF XYZ=1 THEN 6820
3280 REM ###   copy of 'age table'   ###
3290 LPRINT
3300 S0$=CHR$(27)+"S"+CHR$(0):
3310 S1$=CHR$(27)+"T"
3320 WIDTH 80,25
3330 LPRINT USING"   @  (@)  ";S$,SM$
3340 LPRINT USING "    ζ(@) = ####.#  ";ST$,Z
3350 LPRINT USING "    ρ(@) =##.####x10@6@ cm@-2@ (N =#####)";ST$,TD,
S0$,S1$,S0$,S1$,ND
3360 LPRINT STRING$(75,"-")
3370 LPRINT "sample   spontaneous tracks";TAB(32);"induced tracks";TA
B(52);"age"
3380 LPRINT " no.";TAB(10);"number";TAB(20);"density";TAB(30);"number
";TAB(40);"density";TAB(51);"Ma (± 1σ)
3390 LPRINT USING"                    x10@6@ cm@-2@          x10@6@
cm@-2@";S0$,S1$,S0$,S1$,S0$,S1$,S0$,S1$
3400 LPRINT STRING$(75,"-")
3410 FOR I=1 TO N
3420 LPRINT USING" ##      #####    ####.####  #####   ####.####  ###
#.### ±###.###";I,A(I),PS(I),C(I),PI(I),T(I),E(I)
3430 NEXT I
3440 LPRINT STRING$(75,"-")
3450 LPRINT :LPRINT
3460 '
3470 A=0:B=0:C=0:E=0
3480 FOR I=1 TO N
3490 A=A+A(I):B=B+B(I):C=C+C(I):E=E+D(I):PS=A/B:PI=C/E
3500 NEXT I
3510 T=10^4*LOG(1+1.55125*10^(-4)*Z*PS*TD/PI)/1.55125
3520 IF QS=1 THEN T=10^4*LOG(1+1.55125*10^(-4)*Z*PS*TD/(PI*2))/1.5512
5
3530 F=T*SQR(1/A+1/C+1/ND)
3540 LPRINT USING" total   #####    ####.####  #####   ####.####  ###
#.### ±###.###";A,PS,C,PI,T,F
3550 LPRINT
3560 LPRINT USING "  correlation coefficient =##.### ";CC
3570 LPRINT
3580 IF QS=2 THEN LPRINT " spontaneous track surface  : 2π ":LPRINT
:LPRINT
```

```
3590 INPUT"  1) continue    2) finish    ";A
3600 IF 2=A THEN END
3610 CLS 3:GOTO 2080
3620 '
3630 REM ###   copy of age or zeta histogram   ###
3640 IF XYZ=1 THEN 7120
3650 INPUT "  Width of age =    ";A
3660 A$=CHR$(27)+"."
3670 LPRINT A$+"cls"
3680 LPRINT A$+"locate (5,26)"
3690 LPRINT USING " @ (@) ";S$,SM$
3700 LPRINT A$+"line (126,1100)-(1726,2700),,b"
3710 FOR I=1 TO 19 :J=I*80+126
3720 LPRINT USING A$+"line (#### ,2700)-(#### ,2680)";J,J
3730 NEXT I
3740 FOR I=1 TO 3 :J=400*I+126
3750 LPRINT USING A$+"line (#### ,2700)-(#### ,2660)";J,J
3760 NEXT I
3770 FOR I=0 TO 4 :J=15*I+3:K=5*I*A
3780 LPRINT USING A$ +"locate (### ,62)";J
3790 LPRINT USING "####";K
3800 NEXT I
3810 FOR I=1 TO 4 : J=60-9*I : K=5*I
3820 LPRINT USING A$+"locate (2,##)";J
3830 LPRINT USING "##";5*I
3840 L=2700-400*I
3850 LPRINT USING A$+"line (126,####)-(166,####)";L,L
3860 NEXT I
3870 IF XYZ=1 THEN 7140
3880 MAX=T(1)
3890 FOR I=2 TO N
3900 IF MAX < T(I) THEN MAX=T(I)
3910 NEXT I
3920 G=INT(MAX/A)+1
3930 FOR I=1 TO G
3940 CA(I)=0
3950 NEXT I
3960 FOR I=1 TO N
3970 F=INT(T(I)/A)+1
3980 CA(F)=CA(F)+1
3990 NEXT I
4000 FOR I=1 TO G
4010 J=I+1:K=46+80*I:L=36+80*J:M=2700-CA(I)*80
4020 LPRINT USING A$+"line (#### ,2700)-(#### ,####),,b";K,L,M
4030 NEXT I
4040 IF XYZ=1 THEN GOTO 7280
4050 LPRINT A$+"locate (69,62)":LPRINT "(Ma)"
4060 LPRINT A$+"locate (0,65)"
4070 LPRINT A$+"cls"
4080 PRINT "      1) continue       2) finish  ?"
4090 INPUT A
4100 IF 2=A THEN END
4110 CLS 3:GOTO 2080
4120 '
4130 REM ### copy of 'log ρs - log ρi diagram' ###
4140 FOR I=1 TO N
4150 IF A(I)=0 THEN PRINT "Not suitable for log ρs - log ρi diagram"
4160 NEXT I
```

```
4170 FOR I=1 TO N
4180 IF A(I)=0 THEN GOTO 4480
4190 NEXT I
4200 A$=CHR$(27)+"."
4210 LPRINT A$+"locate (8,26)"
4220 LPRINT USING " @ (@) ";S$,SM$
4230 LPRINT A$+"line (200,2700)-(1800,2700)"
4240 LPRINT A$+"line (200,2700)-(200,1100)"
4250 FOR I=1 TO 3
4260 A=200+500*I:B=7+19*I:C=4+I
4270 LPRINT USING A$+"line (#### ,2700)-(#### ,2660)";A,A
4280 LPRINT USING A$+"locate (## ,62)";B
4290 LPRINT USING "#";C
4300 NEXT I
4310 FOR I=1 TO 3
4320 A=2700-500*I:B=61-11*I:C=4+I
4330 LPRINT USING A$+"line (200,####)-(240,####)";A,A
4340 LPRINT USING A$+"locate (6,##)";B
4350 LPRINT USING "#";C
4360 NEXT I
4370 LPRINT A$+"locate (6,62)"
4380 LPRINT "4"
4390 FOR I=1 TO N
4400 X(I)=200+500*(LOG(PI(I)*10^6)/LOG(10)-4)
4410 Y(I)=2700-500*(LOG(PS(I)*10^6)/LOG(10)-4)
4420 LPRINT USING A$+"circle (#### ,####),15";X(I),Y(I)
4430 NEXT I
4440 LPRINT A$+"locate (10,64)"
4450 LPRINT USING " correlation coefficient = ##.###";CC
4460 LPRINT A$+"locate (0,65)"
4470 LPRINT A$+"cls"
4480 PRINT
4490 PRINT  " 1) continue    2) finish ?  "
4500 INPUT A
4510 IF 2=A THEN END
4520 CLS 3:GOTO 2080
4530 '
4540 REM ###  ρs-ρi diagram   ###
4550 MAXI=PI(1):MAXS=PS(1)
4560 FOR I=2 TO N
4570 IF MAXI<PI(I) THEN MAXI=PI(I)
4580 IF MAXS<PS(I) THEN MAXS=PS(I)
4590 NEXT I
4600 SCREEN 3:CONSOLE,,0:WIDTH 80,25:CLS 3
4610 WINDOW (-10,-120)-(120,10)
4620 VIEW (160,0)-(520,360)
4630 LINE (0,0)-(110,0):LINE (0,0)-(0,-110)
4640 A=INT(MAXI)+1:B=INT(MAXS)+1
4650 C=100/A:D=100/B
4660 FOR I=1 TO N
4670 PIC(I)=PI(I)*C:PSD(I)=-PS(I)*D
4680 PIA(I)=PIC(I)*(1+SQR(1/C(I))):PIB(I)=PIC(I)*(1-SQR(1/C(I)))
4690 PID(I)=PIC(I)+1:PIE(I)=PIC(I)-1
4700 IF A(I)=0 THEN PSA(I)=PSD(I):PSB(I)=PSD(I):GOTO 4720
4710 PSA(I)=PSD(I)*(1+SQR(1/A(I))):PSB(I)=PSD(I)*(1-SQR(1/A(I)))
4720 PSE(I)=PSD(I)+1:PSF(I)=PSD(I)-1
4730 NEXT I
4740 LINE (100,0)-(100,-2)
```

```
4750 LOCATE 58,22:PRINT A
4760 LINE(0,-100)-(2,-100)
4770 LOCATE 18,3:PRINT B
4780 FOR I=1 TO N
4790 LINE (PIB(I),PSD(I))-(PIA(I),PSD(I))
4800 LINE (PIB(I),PSE(I))-(PIB(I),PSF(I))
4810 LINE (PIA(I),PSE(I))-(PIA(I),PSF(I))
4820 LINE (PIC(I),PSA(I))-(PIC(I),PSB(I))
4830 LINE (PIE(I),PSA(I))-(PID(I),PSA(I))
4840 LINE (PIE(I),PSB(I))-(PID(I),PSB(I))
4850 NEXT I
4860 AO=0:BO=0:CO=0:DO=0
4870 FOR I=1 TO N
4880 AO=AO+PI(I):BO=BO+PS(I):CO=CO+PI(I)*PS(I):DO=DO+PI(I)*PI(I)
4890 NEXT I
4900 P=(AO*BO-N*CO)/(AO*AO-N*DO):Q=(BO-P*AO)/N:
4910 P=-P*D/C:Q=-Q*D:R=Q+P*100
4920 LINE (0,Q)-(100,R),1
4930 S=-CO*D*100/(DO*C)
4940 LINE (0,0)-(100,S),4
4950 LOCATE 19,22:PRINT 0
4960 LOCATE 30,2:PRINT USING" @ ";S$
4970 LOCATE 25,1:INPUT "Width of ρi = ? ",E
4980 F=INT(A/E)
4990 FOR I=1 TO F
5000 G=100/A*E*I
5010 LINE(G,0)-(G,-2)
5020 H=19+INT(39/A*E*I):J=E*I
5030 LOCATE H,22:PRINT USING "##.#";J
5040 NEXT I
5050 LOCATE 50,1:INPUT "Width of ρs = ? ",K
5060 L=INT(B/K)
5070 FOR I=1 TO L
5080 M=-100/B*K*I
5090 LINE (0,M)-(2,M)
5100 O=22-INT(19/B*K*I):P=K*I
5110 LOCATE 18,O:PRINT USING "##.#";P
5120 NEXT I
5130 PRINT :PRINT USING"   cc = #.###";CC
5140 LOCATE 10,23:PRINT " 1) continue    2) finish ?  "
5150 INPUT A
5160 IF 2=A THEN END
5170 CLS 3:GOTO 2080
5180 '
5190 REM  ###   copy of 'ρs-ρi diagram'  ###
5200 LOCATE 20,6:INPUT"width of ρi (x10^6 /cm^2) = ? ",E
5210 LOCATE 20,10:INPUT "width of ρs (x10^6 /cm^2) = ? ",K
5220 MAXI=PI(1):MAXS=PS(1)
5230 FOR I=2 TO N
5240 IF MAXI<PI(I) THEN MAXI=PI(I)
5250 IF MAXS<PS(I) THEN MAXS=PS(I)
5260 NEXT I
5270 A$=CHR$(27)+"."
5280 LPRINT A$+"locate (10,22)"
5290 LPRINT USING " @ (@) ";S$,SM$
5300 LPRINT A$+"line (200,2700)-(1800,2700)"
5310 LPRINT A$+"line (200,2700)-(200,1100)"
5320 A=INT(MAXI)+1:B=INT(MAXS)+1
```

```
5330 C=1500/A:D=1500/B
5340 FOR I=1 TO N
5350 PIC(I)=200+PI(I)*C:PSD(I)=2700-PS(I)*D
5360 PIA(I)=200+PI(I)*C*(1+SQR(1/C(I)))
5370 PIB(I)=200+PI(I)*C*(1-SQR(1/C(I)))
5380 PID(I)=PIC(I)+10:PIE(I)=PIC(I)-10
5390 IF A(I)=0 THEN PSA(I)=2700-PS(I)*D:PSB(I)=2700-PS(I)*D:GOTO 5420
5400 PSA(I)=2700-PS(I)*D*(1+SQR(1/A(I)))
5410 PSB(I)=2700-PS(I)*D*(1-SQR(1/A(I)))
5420 PSE(I)=PSD(I)+10:PSF(I)=PSD(I)-10
5430 NEXT I
5440 FOR I=1 TO N
5450 LPRINT USING A$+"line (#### ,####)-(#### ,####)";PIB(I),PSD(I),P
IA(I),PSD(I)
5460 LPRINT USING A$+"line (#### ,####)-(#### ,####)";PIB(I),PSE(I),P
IB(I),PSF(I)
5470 LPRINT USING A$+"line (#### ,####)-(#### ,####)";PIA(I),PSE(I),P
IA(I),PSF(I)
5480 LPRINT USING A$+"line (#### ,####)-(#### ,####)";PIC(I),PSA(I),P
IC(I),PSB(I)
5490 LPRINT USING A$+"line (#### ,####)-(#### ,####)";PIE(I),PSA(I),P
ID(I),PSA(I)
5500 LPRINT USING A$+"line (#### ,####)-(#### ,####)";PIE(I),PSB(I),P
ID(I),PSB(I)
5510 NEXT I
5520 F=INT(A/E)
5530 FOR I=1 TO F
5540 G=200+1500/A*E*I:H=7+INT(57/A*E*I):J=E*I
5550 LPRINT USING A$+"line (#### ,2700)-(#### ,2660)";G,G
5560 LPRINT USING A$+"locate (## ,62)";H
5570 LPRINT USING "##.#";J
5580 NEXT I
5590 L=INT(B/K)
5600 FOR I=1 TO L
5610 M=2700-1500/B*K*I:O=61-INT(35/B*K*I):P=K*I
5620 LPRINT USING A$+"line (200,####)-(240,####)";M,M
5630 LPRINT USING A$+"locate (4,##)";O
5640 LPRINT USING "##.#";P
5650 NEXT I
5660 LPRINT A$+"locate (6,61)"
5670 LPRINT "0"
5680 AO=0:BO=0:CO=0:DO=0
5690 FOR I=1 TO N
5700 AO=AO+PI(I):BO=BO+PS(I):CO=CO+PI(I)*PS(I):DO=DO+PI(I)*PI(I)
5710 NEXT I
5720 P=(AO*BO-N*CO)/(AO*AO-N*DO):Q=(BO-P*AO)/N
5730 P=-P*D/C:Q=2700-Q*D:R=Q+1500*P
5740 IF Q>2700 THEN GOTO 5760
5750 LPRINT USING A$+"line (200,####)-(1700,####)";Q,R:GOTO 5790
5760 BB=Q:DD=R
5770 QQ=(2700-BB+2*(DD-BB)/15)*1500/(DD-BB)
5780 LPRINT USING A$+"LINE (#### ,2700)-(1700,####)";QQ,R
5790 S=2700-CO*D*1500/(DO*C)
5800 LPRINT USING A$+"line (200,2700)-(1700,####),,,&heeee";S
5810 LPRINT A$+"locate (10,64)"
5820 LPRINT USING "correlation coefficient = ##.###";CC
5830 LPRINT A$+"locate (0,65)"
5840 LPRINT A$+"cls"
```

```
5850 PRINT
5860 PRINT " 1) continue    2) finish ?  "
5870 INPUT A
5880 IF 2=A THEN END
5890 CLS 3:GOTO 2080
5900 '
5910 REM ### list of input data ###
5920 PRINT USING "     @     ";S$
5930 IF XYZ=1 THEN 7320
5940 PRINT USING "     N (@) =##### ,     A (@) =#### (x100) ";ST$,ND,
ST$,CAS:PRINT
5950 FOR I=1 TO N
5960 GS(I)=B(I)/AG:GI(I)=D(I)/AG
5970 PRINT USING "  Ns =#### , As =### ,      Ni =#### , Ai =###  ";A(
I),GS(I),C(I),GI(I)
5980 NEXT I :PRINT :PRINT
5990 INPUT "   1) continue     2) finish ?   ",A
6000 IF 2=A THEN END
6010 CLS 3:GOTO 2080
6020 '
6030 REM ### copy of 'list of input data '  ###
6040 LPRINT USING "    @    ";S$
6050 IF XYZ=1 THEN 7350
6060 LPRINT USING "    N (@) =##### ,     A (@) =#### (x100)  ";ST$,ND
,ST$,CAS:LPRINT
6070 FOR I=1 TO N
6080 GS(I)=B(I)/AG:GI(I)=D(I)/AG
6090 LPRINT USING "  Ns =#### , As =### ,     Ni =#### , Ai =###  ";A(
I),GS(I),C(I),GI(I)
6100 NEXT I:LPRINT :LPRINT
6110 INPUT "  1) continue     2) finish ?   ",A
6120 IF 2=A THEN END
6130 CLS 3:GOTO 2080
6140 CLS 3:LOCATE 5,2:INPUT"  Sample code of age standard (B:file nam
e) ?  ",S$
6150 LOCATE 5,4:INPUT"  Reference age of standard (Ma) ? ",TS
6160 LOCATE 5,10:INPUT"  Name of dosimeter glass ? ",SG$
6170 LOCATE 5,12:INPUT"  Counted track number of dosimeter glass ? ",
NIG
6180 LOCATE 5,14:INPUT"  Counted area of dosimeter glass (x100 grids)
 ? ",AIG
6190 LOCATE 5,16:INPUT"Your sample ? --- 1) zircon  2) apatite  3) sp
hene ",SM
6200 IF SM=1 THEN SM$="zircon"
6210 IF SM=2 THEN SM$="apatite"
6220 IF SM=3 THEN SM$="sphene"
6230 CLS 3:GOTO 1260
6240 '
6250 REM ### zeta calibration ###
6260 AIG=AIG*AG*100
6270 PSG=NIG/AIG
6280 FOR I=1 TO N
6290 Z(I)=(EXP(1.55125*10^(-4)*TS)-1)*C(I)*B(I)*AIG/(1.55125*10^(-4)*
A(I)*D(I)*NIG)
6300 IF QS=1 THEN Z(I)=Z(I)*2
6310 EZ(I)=Z(I)*SQR(1/A(I)+1/C(I)+1/NIG)
6320 NEXT I
6330 CLS 3:GOTO 1950
```

```
6340 '
6350 LOCATE 5,4:PRINT "   1) zeta table   2) zeta histogram   3) log
ρs - log ρi diagram "
6360 GOTO 2110
6370 '
6380 REM ### zeta table ###
6390 WIDTH 80,25
6400 PRINT USING "    @  (@)      T = ####.## (Ma)";S$,SM$,TS
6410 PRINT USING "    @    ρ(@) =##.#### x 10^4 cm^(-2)  (N =#####)"
;SG$,SG$,PSG,NIG
6420 PRINT STRING$(75,"-")
6430 PRINT "sample   spontaneous tracks";TAB(32);"induced tracks";TAB
(52);"zeta value"
6440 PRINT " no.";TAB(10);"number";TAB(20);"density";TAB(30);"number"
;TAB(40);"density";TAB(53);"   (±1σ)"
6450 PRINT STRING$(75,"-")
6460 FOR I=1 TO N
6470 PRINT USING " ###      #####   ####.####  #####    ####.####  ##
##.### ±###.###";I,A(I),PS(I),C(I),PI(I),Z(I),EZ(I)
6480 NEXT I
6490 PRINT STRING$(75,"-")
6500 PRINT :PRINT
6510 '
6520 A=0:B=0:C=0:E=0
6530 FOR I=1 TO N
6540 A=A+A(I):B=B+B(I):C=C+C(I):E=E+D(I)
6550 NEXT I
6560 PS=A/B:PI=C/E
6570 Z=(EXP(1.55125*10^(-4)*TS)-1)*C*B*AIG/(1.55125*10^(-4)*A*E*NIG)
6580 IF QS=1 THEN Z=Z*2
6590 EZ=Z*SQR(1/A+1/C+1/NIG)
6600 PRINT USING " total    #####   ####.####  #####    ####.####  ##
##.### ±###.###";A,PS,C,PI,Z,EZ
6610 PRINT
6620 PRINT USING "  correlation coefficient = #.### ";CC
6630 PRINT
6640 GOTO 2450
6650 '
6660 PRINT "                zeta value histogram  ":PRINT
6670 INPUT "   Width of zeta value = ";A:CLS 3:GOTO 2570
6680 '
6690 MAX=Z(1)
6700 FOR I=2 TO N
6710 IF MAX<Z(I) THEN MAX=Z(I)
6720 NEXT I
6730 G=INT(MAX/A)+1
6740 FOR I=1 TO G
6750 CL(I)=0
6760 NEXT I
6770 FOR I=1 TO N
6780 F=INT(Z(I)/A)+1
6790 CL(F)=CL(F)+1
6800 NEXT I
6810 GOTO 2840
6820 '
6830 REM ### copy of ' zeta table'   ###
6840 LPRINT
6850 S0$=CHR$(27)+"S"+CHR$(0):
```

```
6860 S1$=CHR$(27)+"T"
6870 WIDTH 80,25
6880 LPRINT USING"   @  (@)      T = ####.##  (Ma)";S$,SM$,TS
6890 LPRINT USING "   @     ρ(@) =##.#### x 10@4@ cm@-2@  (N =#####)"
;SG$,SG$,PSG,S0$,S1$,S0$,S1$,NIG
6900 LPRINT STRING$(75,"-")
6910 LPRINT "sample   spontaneous tracks";TAB(32);"induced tracks";TA
B(52);"zeta value"
6920 LPRINT " no.";TAB(10);"number";TAB(20);"density";TAB(30);"number
";TAB(40);"density";TAB(51);"   (± 1σ)"
6930 LPRINT USING"                    x10@6@ cm@-2@          x10@6@
cm@-2@";S0$,S1$,S0$,S1$,S0$,S1$,S0$,S1$
6940 LPRINT STRING$(75,"-")
6950 FOR I=1 TO N
6960 LPRINT USING " ##     #####    ####.####  #####    ####.####  ##
##.### ±###.###";I,A(I),PS(I),C(I),PI(I),Z(I),EZ(I)
6970 NEXT I
6980 LPRINT STRING$(75,"-")
6990 LPRINT :LPRINT
7000 '
7010 A=0:B=0:C=0:E=0
7020 FOR I=1 TO N
7030 A=A+A(I):B=B+B(I):C=C+C(I):E=E+D(I):PS=A/B:PI=C/E
7040 NEXT I
7050 Z=(EXP(1.55125*10^(-4)*TS)-1)*C*B*AIG/(1.55125*10^(-4)*A*E*NIG)
7060 IF QS=1 THEN Z=Z*2
7070 EZ=Z*SQR(1/A+1/C+1/NIG)
7080 LPRINT USING " total  #####    ####.####  #####    ####.####  ##
##.### ±#####.###";A,PS,C,PI,Z,EZ
7090 LPRINT
7100 LPRINT USING "   correlation coefficient = #.### ";CC
7110 LPRINT :GOTO 3580
7120 '
7130 INPUT "  Width of zeta value = ";A:GOTO 3660
7140 '
7150 MAX=Z(1)
7160 FOR I=2 TO N
7170 IF MAX<Z(I) THEN MAX=Z(I)
7180 NEXT I
7190 G=INT(MAX/A)+1
7200 FOR I=1 TO G
7210 CA(I)=0
7220 NEXT I
7230 FOR I=1 TO N
7240 F=INT(Z(I)/A)+1
7250 CA(F)=CA(F)+1
7260 NEXT I
7270 GOTO 4000
7280 '
7290 LPRINT A$+"locate (69,62)":LPRINT "(zeta)"
7300 GOTO 4060
7310 '
7320 BIG=AIG/AG:PRINT USING "     @      N =#####      A =#####     ";
SG$,NIG,BIG
7330 GOTO 5950
7340 '
7350 BIG=AIG/AG:LPRINT USING "    @      N =#####      A =#####     ";
SG$,NIG,BIG
7360 GOTO 6070
```